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DEVELOPMENT OF INEXPENSIVE SURFACE FINISHING PROCESS FOR TRANSPARENT ARMOR

John S. Haggerty, et al

Arthur D. Little, Incorporated

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13. ABSTRACT

The objectives of this program were to develop a process for coating rough-ground sapphire tiles with index matching glasses. The cost of finishing the transparent armor tiles can thereby be reduced by polishing with conventional, inexpensive glass polishing procedures rather than those needed for sapphire. These objectives were met using a lead-silicate glass on pretreated sapphire tiles. The optical properties of the coated tiles were essentially those of sapphire. Alternative finishing procedures based on refiring rough-ground, coated tiles and purely chemical polishing were demonstrated. These could lead to even further cost reduction in finishing transparent armor.

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Technical Report by

John S. Haggerty
Michael Rossetti

Arthur D. Little, Inc.
Cambridge, Massachusetts 02140

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I. INTRODUCTION

The technology required to produce large transparent shapes for transparent armor applications has advanced to a point where the potential use of sapphire single crystal windows can be considered a reality. Now that the required technology has been demonstrated, the critical issue has become one of producing the finished windows at a reasonable cost. Arthur D. Little, Inc., concluded in its final reports to AMMRC (DAAG 46-69-C-0078 and DAAG 46-72-C-0168) that a large fraction of the total cost of a window was associated with surface finishing operations required to give an optically smooth and flat surface. The marked difference between the cost of ground and polished glass (typically of the order of \$1 per ft^2) and sapphire (approximately \$15 per in^2 in 1971 with a projected minimum of \$2-\$3 per in^2) is an indication of the effect finishing processing could have on cost of finished sapphire windows. Prior to undertaking this program, ADL demonstrated that rough ground sapphire tiles could be made transparent by coating them with an inorganic glass with an index of refraction close to that of sapphire. These tiles were then finished by glass polishing procedures. This program was intended to further develop the process and provide samples for ballistic evaluation.

Specifically, the objective of the program was to develop a coating process which demonstrated the optical properties of nominally 1-inch diameter coated sapphire tiles. This laboratory scale optimized process was then to be used on 4-inch diameter tiles intended for ballistic testing. The results of this evaluation would determine whether further development of a coating process for transparent armor was warranted. Our basic approach was one of simplifying the process as much as possible while achieving an optical quality which demonstrated the feasibility of the concept.

II. TILE COATING PROGRAM

A. Requirements for Inorganic Coatings

There are numerous criteria that exposed surfaces must meet, such as dust and rain erosion resistance, durability, etc. We limited our attention to two properties which were critical for meeting our objectives: matching the index of refraction and achieving a compressive stress in the glaze coating by selecting a glass with an appropriate thermal expansion coefficient.

1. Optical Properties

Ideally, a glaze would have the same index and dispersion $[n,(\lambda)]$ as the substrate to eliminate the optical interface between the two media. We considered the effect of an index mismatch to determine how close they must be to one another while still giving acceptable optical performance. Reflection and scattering losses are proportional to $\left(\frac{n_1 - n_2}{n_1 + n_2}\right)^2$ and $1 - n_1/n_2$, respectively: both of which become negligibly small when $n_1 \to n_2$.

Retaining an adequate quality of the transmitted image imposes a more severe restriction on matching the two indices. The angular deflection a light ray undergoes upon crossing the glaze-sapphire interface should not be greater than the eye can resolve, which is approximately 1/2500 radian. Using the geometry shown in Figure 1, according to Snell's Law:

$$
\begin{aligned}
n_1 \sin \theta &= n_2 \sin \phi \\
&= n_2 \sin (\theta - \delta) \\
&= n_2 \sin \theta \cos\delta - n_2 \cos \theta \sin \delta
\end{aligned}
$$

or

$$n_1/n_2 = \cos \delta - \cot \theta \sin \delta$$

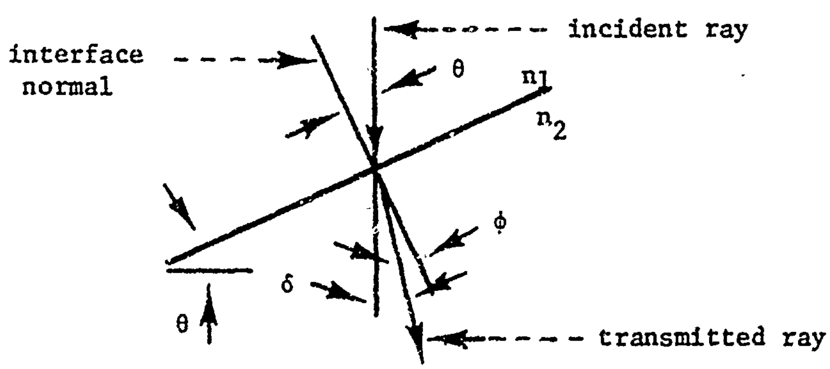


FIGURE 1 GEOMETRY USED TO DETERMINE ANGULAR DEFLECTION OF LIGHT RAY

Solving for the maximum permissible Δn as a function of θ (a measure of the surface roughness of the substrate) with $\delta \leq 1/2500$ radian, one obtains:

θ	Δn_{max}
1°	0.0293
10°	0.0024
45°	0.0005

This shows that the required accuracy of matching the two indices is greatly reduced for surfaces whose features are nearly perpendicular to the transmitted light ray. Thus, from this criteria, the wavelength of surface features is more important than RMS surface roughness (peak height). The natural birefringence of sapphire ($n_{\parallel c}$ = 1.769, $n_{\perp c}$ = 1.760) requires that the uncoated surfaces should not have surface features deviating more than 10° from incident light if the window is to have a better optical quality than the human eye.

Because it was evident that the as-ground surface finishes, as characterized by Scanning Electron Microscopy (SEM), did not meet the 10° criteria, improvement of surface finish by chemical polishing was examined. These procedures and results are discussed later.

2. Thermal Expansion Coefficients

Surprisingly, we were unable to locate mutually consistent thermal expansion coefficient data for sapphire over a temperature range from -196°C to 1000°C. This temperature range included the minimum military service temperature (-60°F) and the set points of all practical glass compositions.

Two samples were taken from orthogonal directions in the $(1\bar{1}02)$ plane; one sample axis was parallel to the $\bar{a}$ axis and the other 90° from it. Thus, these two samples should have exhibited the maximum thermal expansion anisotropy in this plane. The results of the thermal expansion measurements made by Dynatech Corp., Cambridge, Massachusetts, are shown in Figure 2 along with other values reported in the literature. Dynatech observed no difference between the behavior of the samples, within the accuracy of their measurements; thus, it appears reasonable to treat the thermal expansion coefficient in the $(1\bar{1}02)$ plane as isotropic. The thermal expansion coefficient increased approximately linearly from -195 to 250°C and was then essentially constant beyond 400°C. The Dynatech results lie within the range of other reported results.

These results indicate that the glaze should have a mean thermal expansion coefficient between 6.0 and 7.0 x 10^{-6}°C^{-1} to remain in compression under all anticipated service conditions. More precise definitions of required thermal expansion coefficients were not pursued, since the actual stresses developed in a glaze are a complex function of thermal history in the glass transition region.

3. Glass Selected

Based on these index and thermal expansion coefficient requirements, potentially useful glass compositions were investigated.

Several glasses were identified in Morey[1] which match these requirements adequately. One corresponded to a commercially available Ferro #3403 frit; therefore, it was selected. This glass consisted of:

1.4 w/o K_2O	0.3 w/o Na_2O	0.1 w/o CaO
67.8 w/o PbO	2.3 w/o Al_2O_3	28.1 w/o SiO_2

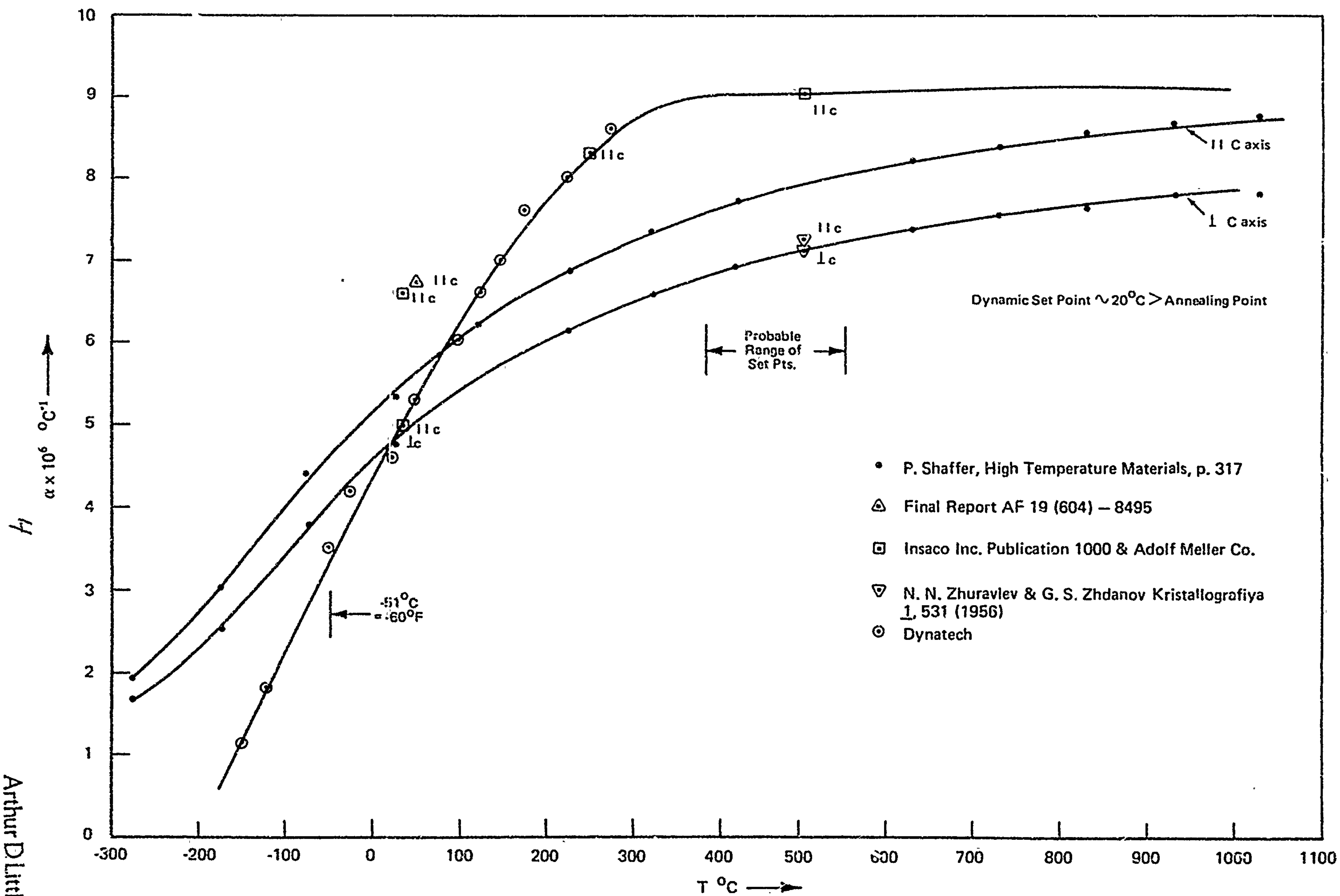


FIGURE 2 REPORTED AND THERMAL EXPANSION MEASUREMENTS OF SAPPHIRE

The index of refraction is in the range of 1.73 to 1.80 and the thermal expansion coefficient is approximately $7.0 \times 10^{-6} {}^\circ C^{-1}$. Closer matches could have been realized; however, it seemed best to work with a commercially available material until any deficiencies were identified.

B. Polishing Processes Used for Tile Pretreatment

Verneuil sapphire boules were sectioned parallel to $(1\bar{1}02)$ planes to produce working samples for chemical polishing optimization and subsequent 1-inch diameter coating processes. The surfaces were ground to a 60-micro inch finish with a diamond wheel using a water soluble oil lubricant. A representative as-ground surface is shown in the SEM photomicrograph in Figure 3.

A grinding track is evident in this particular area. It is obvious that the as-ground surface does not meet the flatness criteria developed previously for required optical performance. It generally consists of rough steps with cleavage type fracture features.

This rough surface presented another problem for achieving good quality coatings; namely, displacing gas from the crevices. Long times at high temperature required to displace trapped gas was not believed to be a practical approach, since all potential glasses tend to dissolve Al_2O_3. Dissolution of excessive Al_2O_3 into the glass would change both the index of refraction and the thermal expansion coefficient uncontrollably near the glass-sapphire interface.

Pretreatments were investigated with the purpose of smoothing the sapphire surfaces prior to coating them with glass. It is also known that the as-grown surfaces are highly stressed due to work damage which causes a higher than average dissolution rate into the glass coating during the glazing cycle. The pretreatment of the sapphire blanks was intended to produce a low-energy, smooth surface for the subsequent coating processes.

Approximately ten sapphire polishing processes found in the literature were used on 1 cm x 1 cm sapphire samples. Scanning electron microscopy was used as the principal characterization technique for evaluating the effectiveness of the pretreatments. The pretreatments which were investigated, included vacuum and H_2 firing, sodium borate and phosphoric acid chemical polishes, the Freon II (Reg. T.M.) technique reported by Rice, et.al.,[2] as well as the technique which was finally selected. Each of the screened pretreatments modified the surface topography. The highly faceted surface shown in Figure 3 resulted from vacuum firing an as-ground surface. Most of the surfaces were no better than the original as-ground surface with respect to their topography. The best polishing process found, including the severity of the thermal cycle, was:

grind

air anneal for 4 hours at 1500°C

chemical polish 1:1 HPO_4:H_2SO_4 15 minutes at 285°C

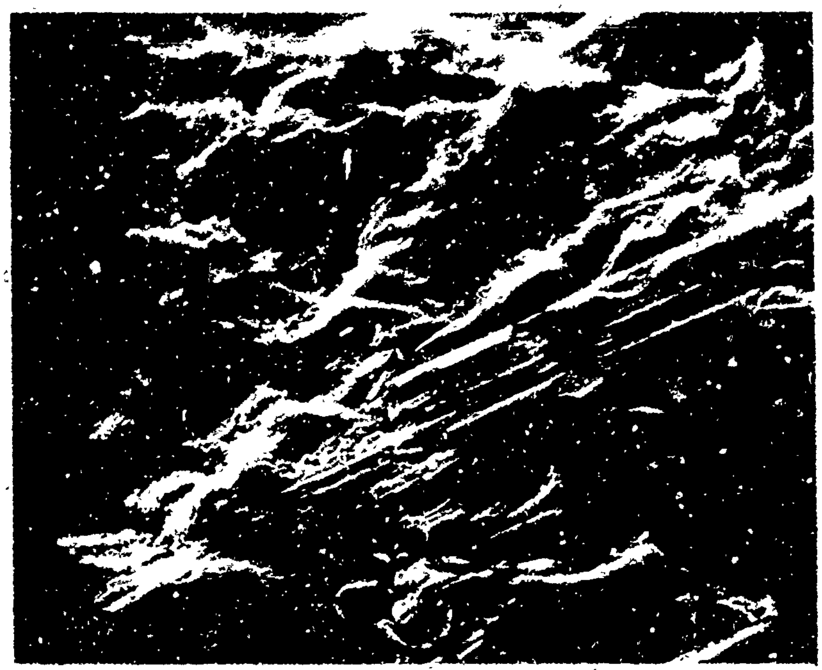


FIGURE 3 As-Ground $(1\bar{1}02)$ Sapphire Surface Observed By Scanning Electron Microscope (SEM) Nominal Magnification 2400X

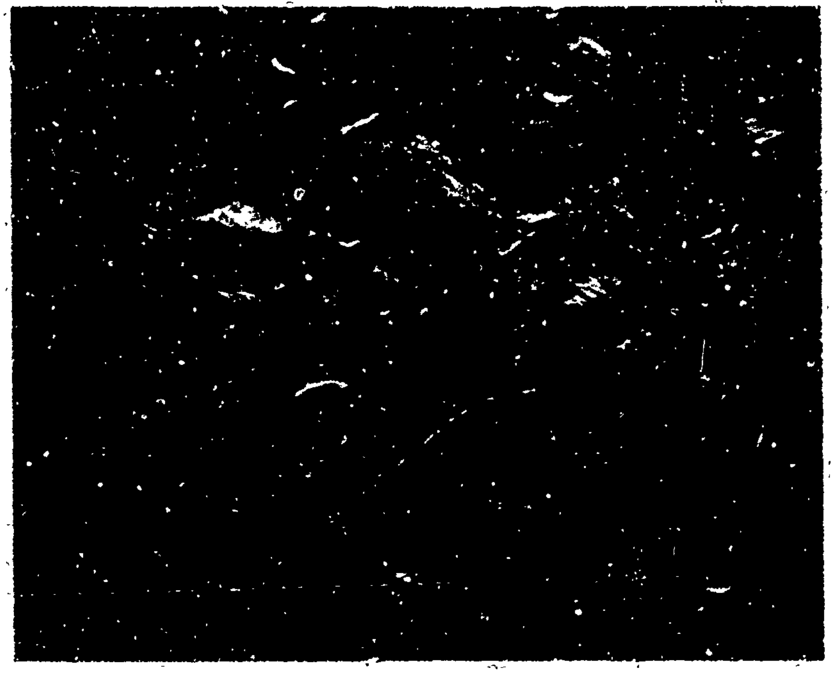


FIGURE 4 $(1\bar{1}02)$ Sapphire Surface Fired in Vacuum
For 2-1/2 Hours at 1570°C
Nominal Magnification 2400X

The surfaces of sapphire after air firing and subsequent chemical polishing are shown in Figures 5 and 6, respectively. A rough, loosely bonded scale forms on the sapphire during air firing. No attempt was made to characterize the scale; however, at locations where the scale flaked off, one could see a smooth surface below the scale. After polishing in the $H_3PO_4:H_2SO_4$ solution, the surfaces appeared as shown in Figure 6. It is probable that most of the polishing occurred during the air annealing cycle and the acid polish only removed the scale.

All of the tiles which were subsequently coated were pretreated by the technique described above.

C. Coating Processes Investigated

The three coating techniques investigated were: melting a powder frit, slumping a premelted glass onto the tiles and dipping the tiles into molten glass.

Dimensionally uniform glaze coatings were produced by the powder process. The coatings frequently contained bubbles which were trapped by impinging frit particle interfaces. The most serious optical defect was a variation of the refractive index across the glass surfaces where frit particles impinged. Lead probably vaporized from the surface of frit particles resulting in locally low indices. Longer firing cycles intended to homogenize the glass, resulted in excessive corrosion of the tiles and devitrification of the glaze. These problems can probably be resolved with different glass compositions; however, we did not have the resources to divert the program to the development of glass compositions.

A second method which consisted of slumping a fired glass sheet onto the tile was investigated. At atmospheric pressure, many bubbles were entrapped between the glass and sapphire. Vacuum slumping eliminated the bubbles but resulted in extensive surface devitrification of the glass. These problems could also have probably been eliminated by modifying the glass composition, since the process is used to bond other glasses.

At the same time as these other two coating techniques were being investigated, the pretreated tiles were dipped edge-on into a glass melt. This process produced pore-free, vitreous coatings which appeared to have acceptable optical and dimensional uniformity. This process also had the advantage that both sides of the tile were coated in a single firing cycle. Within the limited scope of the program, this appeared to be a nearly optimum process.

D. Optimized Coating Cycles

1. One-Inch Diameter Tiles

The furnace and sample insertion-withdrawal mechanism used to coat the 1-inch diameter tiles is shown schematically in Figure 7. It consisted of a vertical, glow-bar heated tube-furnace with a 2-inch internal diameter alumina muffle tube. The bottom of the muffle terminated in a 1/4-inch

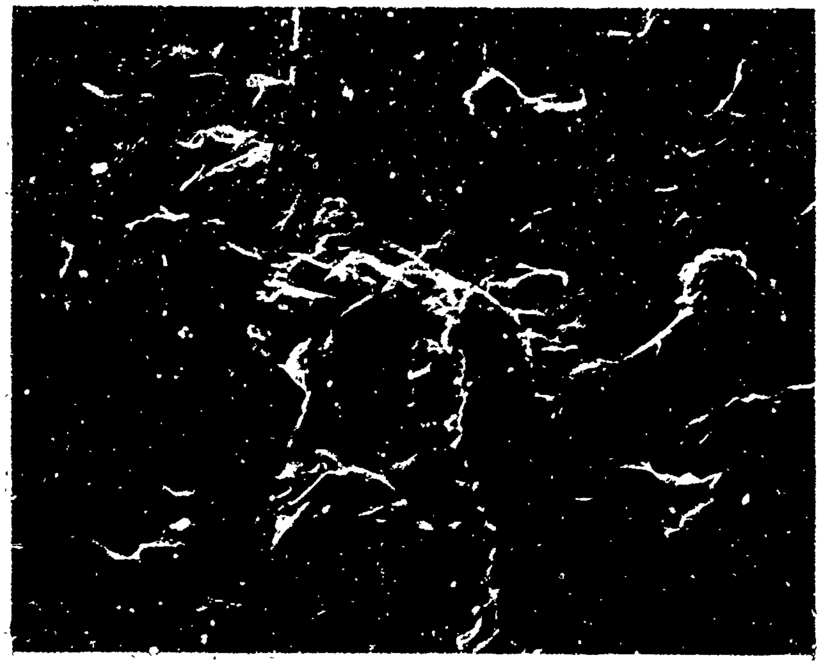


FIGURE 5 (1$\bar{1}$02) Sapphire Surface Fired for 4 Hours
in Air at 1500°C
Nominal Magnification 2400X

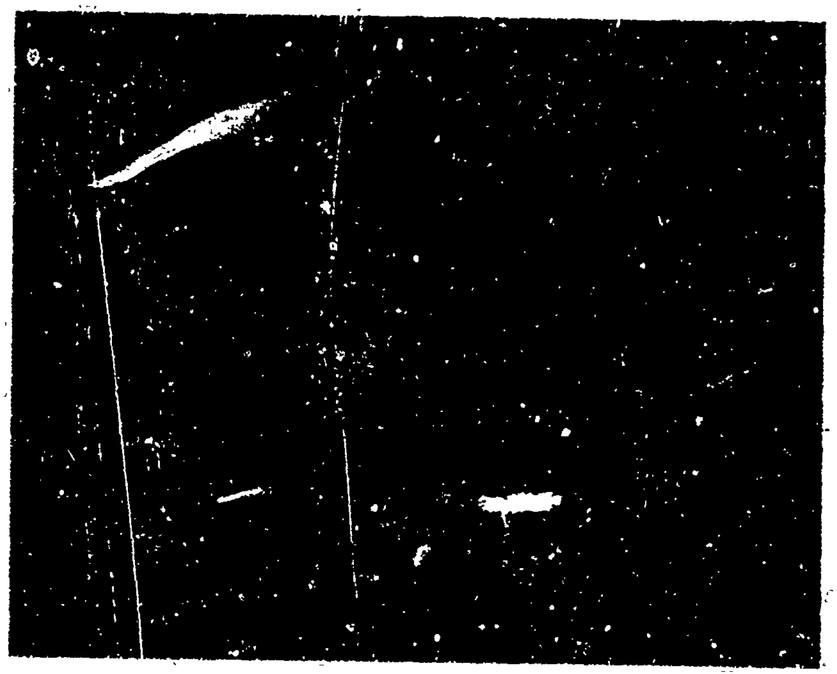


FIGURE 6 ($1\bar{1}02$) Sapphire Surface Fired for 4 Hours In Air at 1500°C Followed by Chemically Polishing For 15 Minutes in 1:1 H_3PO_4:H_2SO_4 at 285°C
Nominal Magnification 1300X

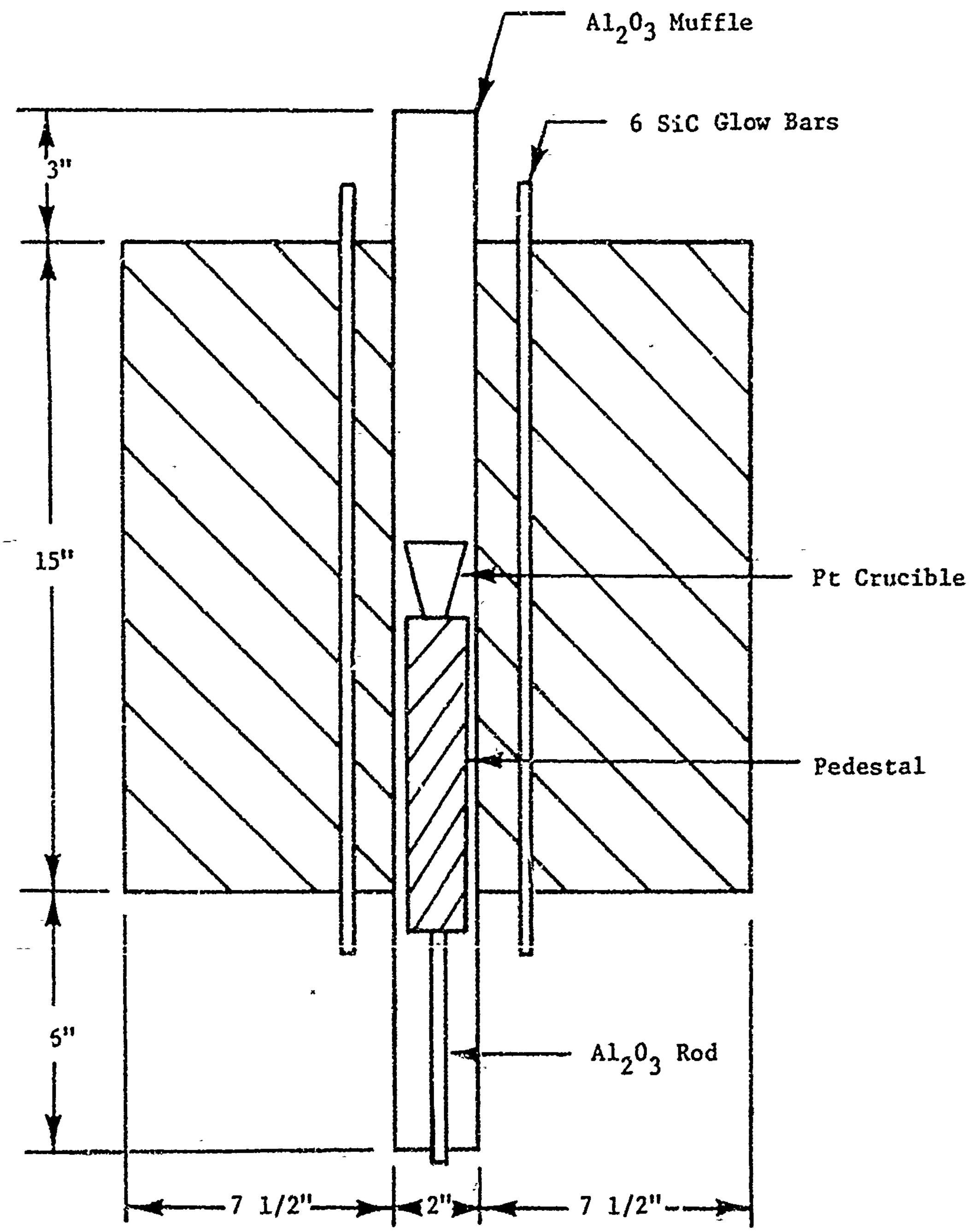


FIGURE 7 Schematic of Furnace For Coating 1-Inch Tiles

internal diameter tubulation. A platinum crucible was supported in the furnace by a movable 1/4-inch diameter Al_2O_3 rod. After the tiles were lowered and soaked just above the glass, they were immersed into the glass by raising the crucible.

The best processing conditions found for the 1-inch diameter tiles were:

lowering time: 20 minutes
soak time above melt: 20 minutes
dip time: 1 minute
drain time: 15 minutes
withdrawal time: 20 minutes

The furnace temperature at the crucible level was 1300°C. The process was conducted in an air atmosphere. Samples were supported by a Pt-10% Rh wire harness. This cycle produced a coating with a minimum thickness between 0.0015 to 0.002 inch on each side of the tiles.

None of the 1-inch diameter, 1/4-inch thick tiles were cracked by thermal shocking. In fact, several tiles which had been cracked during chemical polishing were dipped without any evidence of crack propagation. These results indicated that the coating process could probably be used without modification for the 4-inch diameter tiles.

2. Four-Inch Diameter Tiles

A special furnace was constructed for coating the 4-inch diameter tiles. It is shown schematically in Figure 8.

Like the smaller furnace, this furnace was heated with SiC glow bars. The absence of a muffle proved to be a major difference between the two furnaces, since in the latter case, the glass was exposed directly to the atmosphere which is in equilibrium with the SiC glow bars. Evidently this atmosphere is sufficiently reducing to reduce the lead oxide in this glass at 1300°C.

The furnace consists of the heated area at the bottom, a chimney and, at the top, a cool-down chamber which is closed off from the chimney by a shutter. The furnace was tightly sealed to minimize drafting of inducted air up the chimney.

It was originally intended that Kyanite (Findlay Refractories) crucibles would be utilized in the 4-inch diameter tile dipping furnace. The glass used in this program as well as other lead glasses have been melted in this type of crucible without difficulty in earlier work at ADL. Two of these crucibles broke at temperature allowing the 25-pound glass charges to pour down through the furnace brick work. The glass was eventually contained in the Kyanite crucibles; however, their use was abandoned because of excessive contamination from the crucibles which discolored the glass. The use of alumina crucibles was considered but rejected due to excessive dissolution rates (0.030 inch in 12 hours). Given the glass composition which had been selected, it appeared that platinum was the only obvious choice for a crucible material. A 1-inch by 5-inch by 5-inch, 0.010-inch thick platinum crucible was constructed for this program with

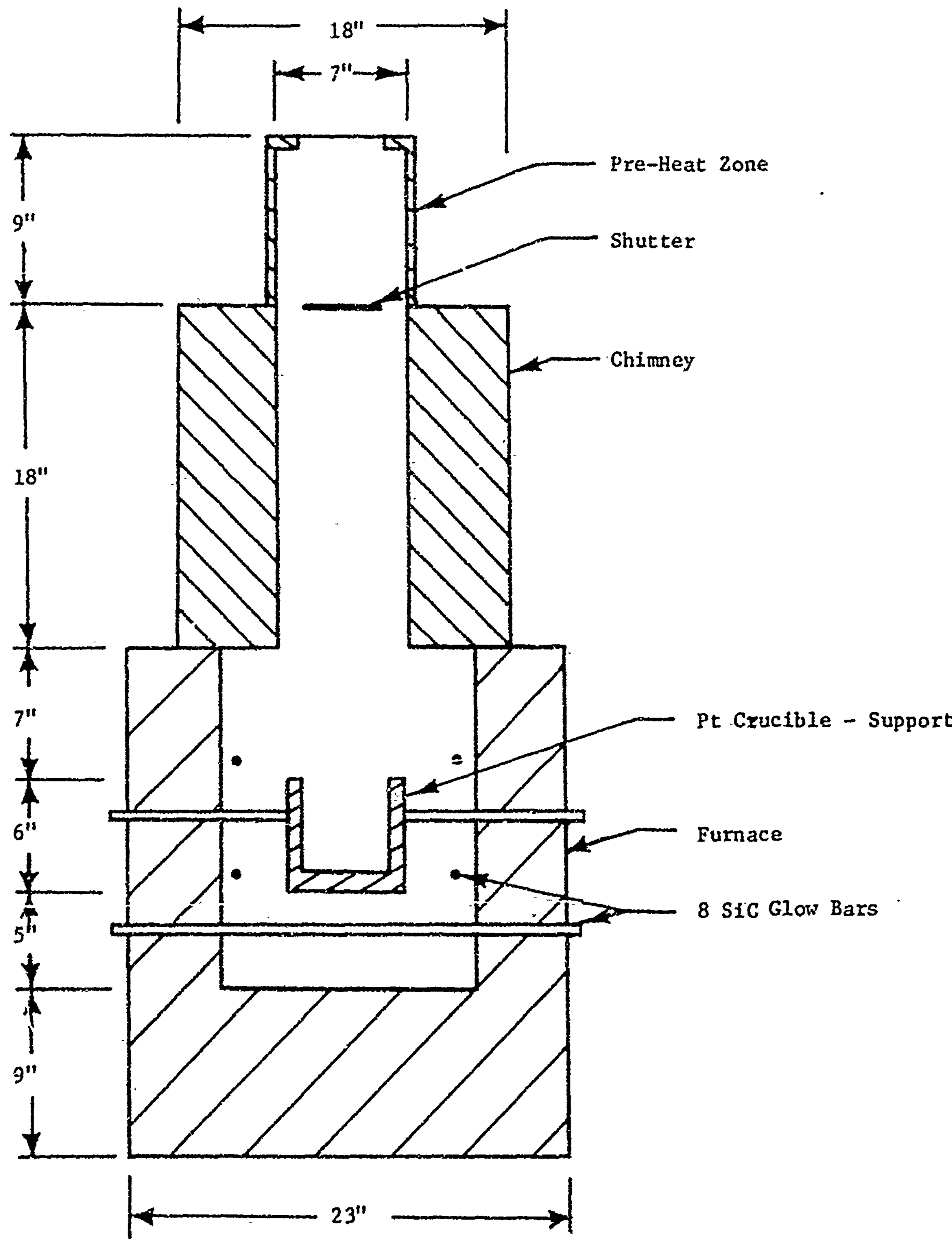


FIGURE 8 Schematic of Furnace For Coating 4-Inch Tiles

ADL funds from ADL-owned platinum. Grain boundaries in the platinum crucible welds were attacked during the first heating in this crucible. This attack was attributed to reduction of the glass by the SiC glow bars. An air lance was then installed into the rebuilt furnace to insure that the atmosphere remained oxidizing. No further problems were encountered with crucible failures.

The glass was stirred between dipping cycles to homogenize the glass. The bulk glass exhibited index variations after each tile was dipped--apparently from dissolved Al_2O_3. Stirring plus a 1-hour soak homogenized the glass adequately.

The following procedures were used to coat the 4-inch tiles. The tiles were suspended 1-inch above the furnace with the shutter closed for 10 to 20 minutes. The tiles reached approximately 150°C in this position. They were then lowered into the upper chamber at 1.5 to 2 inches per minute and held there for 20 minutes with the shutter closed. The temperature of the upper chamber with the shutter closed was approximately 500°C. The shutter was opened half way for 15 minutes and completely for 20 minutes prior to lowering the tiles to a position just above the melt. They were lowered at a rate of 1.8 inches per minute. The tiles were soaked above the glass for 20 minutes, dipped and pulled into the upper chamber at 1.8 inches per minute. The temperature of the tiles when they passed the shutter was approximately 860°C. The tiles were held in the upper chamber with the slit half closed for 10 minutes and fully closed for 20 minutes. The tiles were then pulled out of the chamber and clasped between asbestos pads until they cooled to room temperature. Five of seven tiles cracked during coating (2 tiles cracked on their second dipping cycles) so the process cannot be regarded as optimized.

In all cases, cracking occurred after the tiles passed the shutter slot while they were in the upper chamber. It is not known when they cracked during the 30-minute cooling cycle. It is possible that some drafting of cold air occurred through the upper chamber before the shutter was closed. and packed. When the shutter was stationary, all cracks were packed with Fiber Frax (Re TM) to prevent cold air from being drawn into the upper chamber. When it was moved, e.g., during closing, the Fiber Frax was removed to prevent it from falling into the glass. Thus there was a path for free convection of cold air onto the hot tiles for a short period of time. There did not appear to be any extension of the cracks from the time they were withdrawn from the upper chamber.

Why so many of the glazed tiles cracked is not understood. Several dry runs were made with one of the lower quality tiles with more severe thermal cycles than the one reported above. This tile was never cracked. The final cool-down must be improved before this dipping process is used with other large diameter tiles.

E. Surface Finishing

1. One-Inch Tiles

After the tiles had been coated with glass, the surfaces were polished at ADL and by two vendors. The vendors were utilized primarily to deter-

mine whether they could polish the glass-coated sapphire by conventional polishing techniques.

Both Syncor Co., Cambridge, Mass., and Valpi Co., Holliston, Mass., achieved acceptable polishes on the 1-inch tiles with their polishing processes. The differences between the optical quality of individual tiles were principally caused by index variations in the glass coatings. Both companies indicated that the glass coated tiles could be polished interchangeably with other glass products provided the tiles had a reference surface which was parallel to the sapphire-glass interfaces. The principal reason both polishing houses expressed a need for the reference surface was to avoid polishing through the glass coating into the sapphire. This requirement can be met by applying a thicker and/or a more uniform coating than was achieved. From optical considerations, a small wedge angle in the glass coating should cause no problem.

All of the tiles finished at ADL were polished by conventional hand polishing procedures. Combinations of SiC, Al_2O_3 and diamond grits were used on individual tiles. Our objective was simply one of polishing the tiles as quickly as possible to permit an evaluation of the glass coatings rather than optimizing the polishing process.

2. Four-Inch Tiles

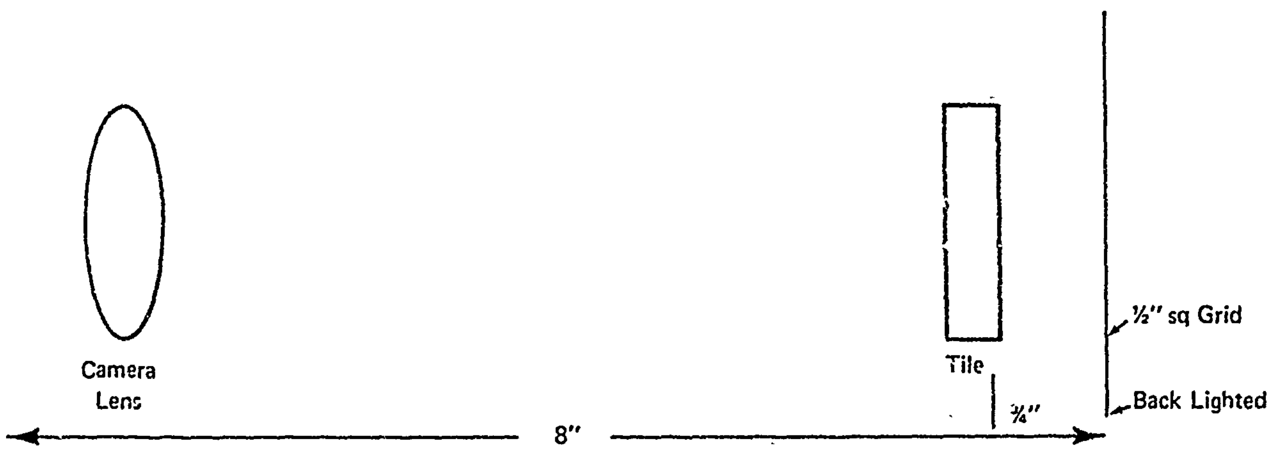
The two tiles which remained in tact after coating were hand polished at ADL. The coating thickness on each tile was carefully mapped so the surfaces could be polished parallel to the sapphire-glass interfaces by using shims. These coated tiles were ground flat on glass lapps with SiC and then finish polished with diamond. The coating thicknesses on each side of the tiles were 0.0015 to 0.0020 inch.

Reasonably good surface finishes were produced on 1-inch tiles by refiring coated tiles which had been rough gorund with SiC. The optical quality of these tiles was not as good as polished tiles; however, they were adequate to demonstrate the feasibility of the process and would provide a smooth surface for ballistic testing. Relatively rapid cooling was necessary to prevent devitrification of the glass coating. It was found that passing the 1-inch tiles through the time-temperature cycle used for dipping resulted in uniform, smooth, vitreous coatings. Two of the coated 4-inch tiles were cracked attempting to finish them by this technique, so the two remaining tiles were hand polished. Complete elimination of finish polishing appears feasible; however, there can be no loss of tiles by thermal shocking if the process is to be considered cost effective.

F. Characteristics of Coated Tiles

1. Optical Properties

The optical characteristics of the tiles evaluated at ADL were: (1) image distortion by photographing a grid through the tile as well as qualitatively by photographing objectives through tiles and (2) in-line transmission. Haze is to be evaluated at AMMRC.



Photographs taken at f32, exposure 45 seconds
Film-High Contrast Polaroid (RTM) Type 51

FIGURE 9 GEOMETRY USED FOR GRID DISTORTION TESTS

a) Image Distortion

It was originally intended that image distortion would be evaluated by photographing a grid with and without a tile between the lens and grid. The double exposure technique would permit evaluation of the window blanks by the uniformity of the line shift in the two exposures. We found that, in practice, a single exposure through the tiles revealed the defective areas. This was felt adequate for this program, since there was no standardized geometry for this testing procedure and no procedure for evaluating the information quantitatively. The geometry used for this technique is shown in Figure 9.

The transmitted images of two small tiles are shown in Figures 10a and 10b. The tile in 10a is in the as-dipped condition. Distortion near the edges is caused by relatively thick coatings in these regions. After polishing, the transmitted images were generally like that shown in Figure 10b. In the central regions, the imaging quality was generally good. Near the edges a small scale distortion was usually evident. The distortion had the appearance of that resulting from an "orange peel" surface; however, interference microscopy indicated that these tiles had smooth, flat surfaces. It was concluded that the optical distortion resulted from non-homogeneous glass coatings. Dissolution of the Al_2O_3 tiles by the glass probably caused the localized index variations.

A qualitative demonstration of the imaging quality of the tiles is shown in Figures 11, 12 and 13. The tiles were approximately 4 inches from the scale and the camera approximately 20 inches from the tiles. Figure 11 shows the condition of the tiles in the as-ground, uncoated condition. The mat surface scatters light to the extent that the lettering on the scale is not distinguishable. Figure 12 shows the optical quality achieved by rough grinding the coated tiles with 600 mesh SiC and refiring the glaze to produce the equivalent of a flame polished surface. Even though this tile cracked during the second heat cycle, the overall quality is still revealed. The surface of the tile is smooth. Variations in coating thickness produce some image distortion. Polishing the coated tiles produces surfaces as shown in Figure 13. The imaging quality of these tiles is generally good. There were some defects near the edges similar to those shown in Figure 10b.

We made no quantitative measurements of the imaging quality of these coated tiles. They appeared to be about the same optical quality as unground window glass. Dimensional and compositional tolerances are higher for the sapphire tiles than for typical window glasses, since their refractive indices are higher.

b) Spectral Transmittance

The fraction of the incident light transmitted ($\overset{*}{T}$)[3] through a dielectric slab with plane parallel surfaces is given by

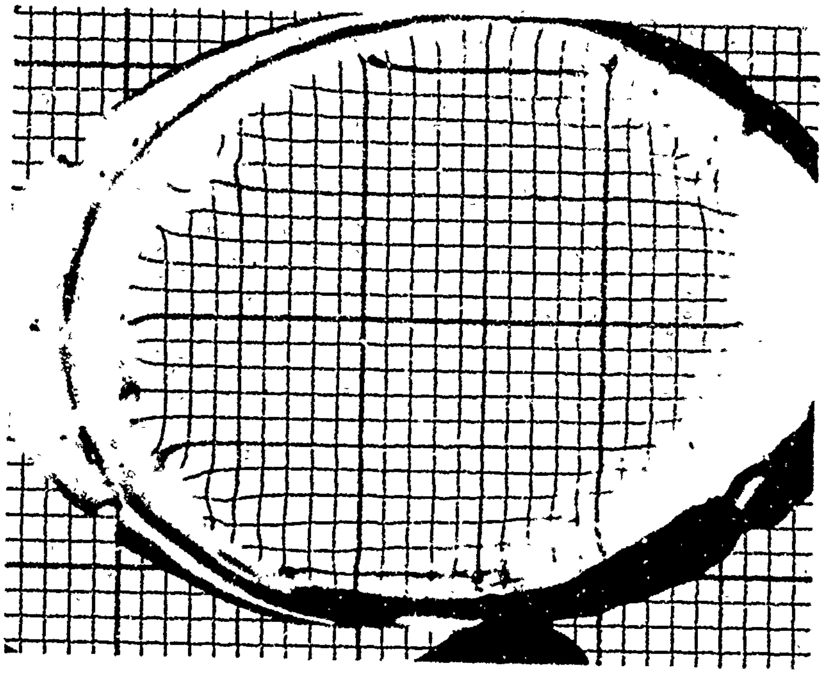


Figure 10a As-Dipped Tile

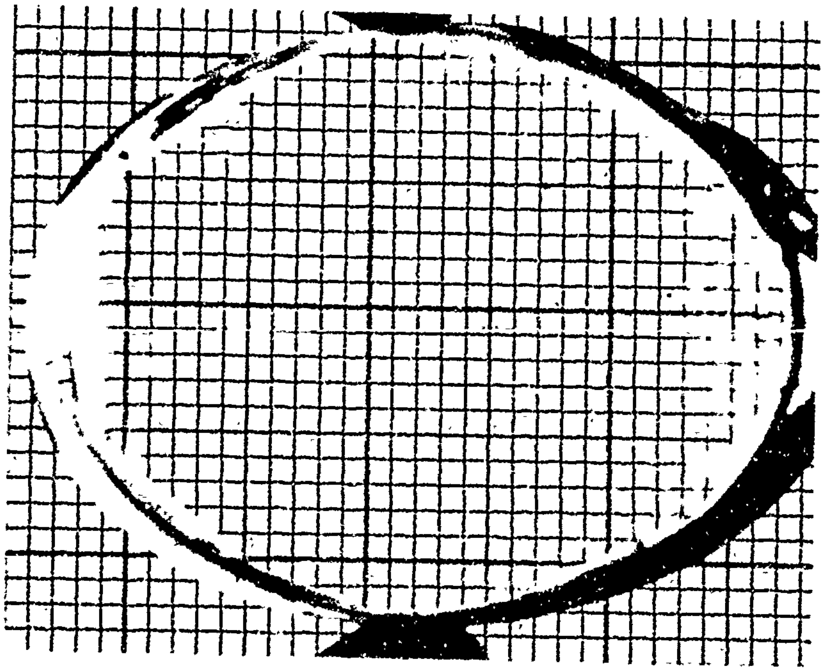


Figure 10b Coated and Polished Tile

Transmitted Images of Grid Photographed Through 1/4-Inch Thick Sapphire Tiles. Grid is 10 Lines Per Inch.

FIGURE 11 Photograph of Scale Taken Through Uncoated, As-Ground Tile

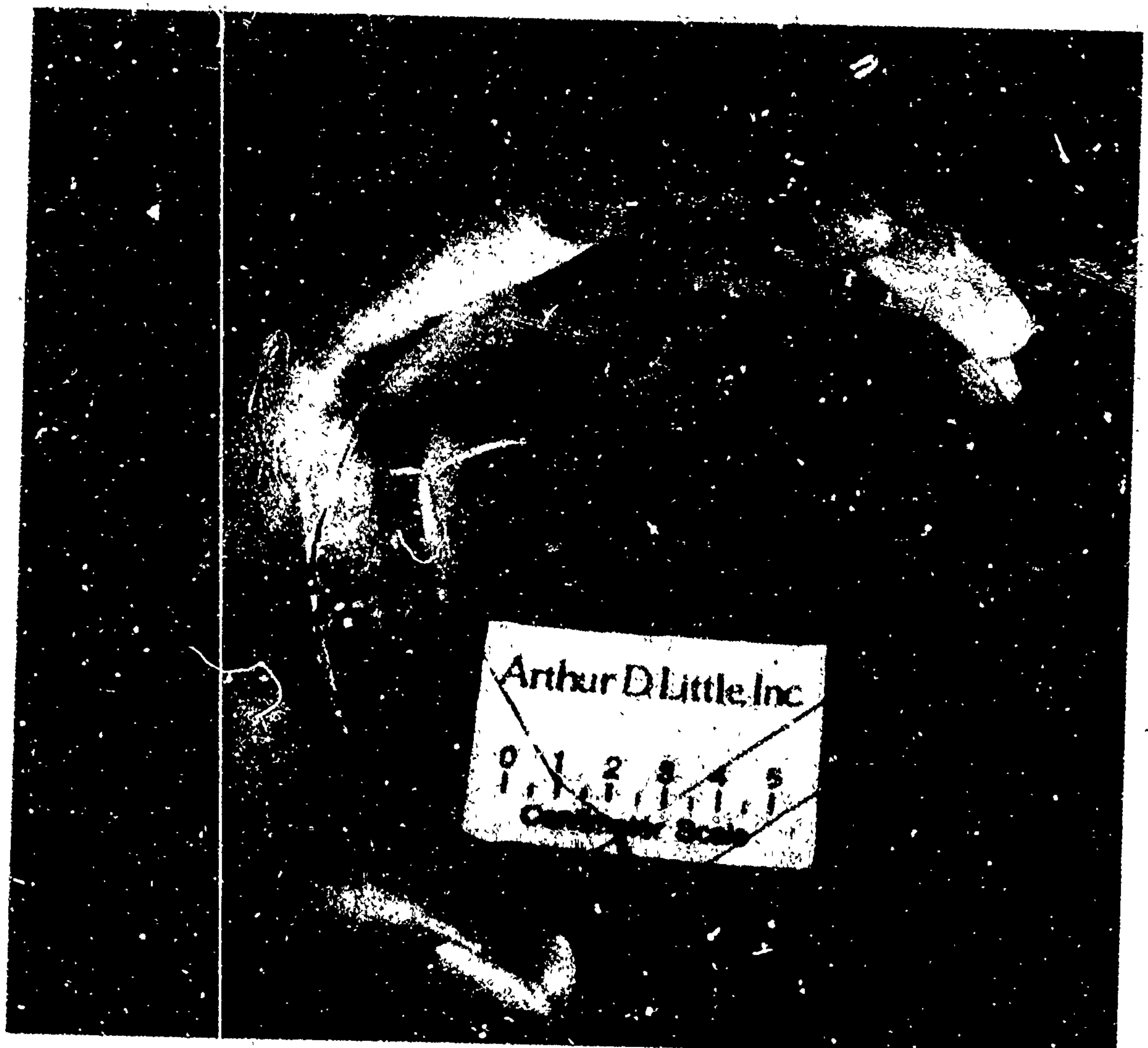


FIGURE 12 Tile Which Had Been Coated, Rough Polished With 600 Mesh SiC and Heat Treated. See Text For Heat Treatment Details

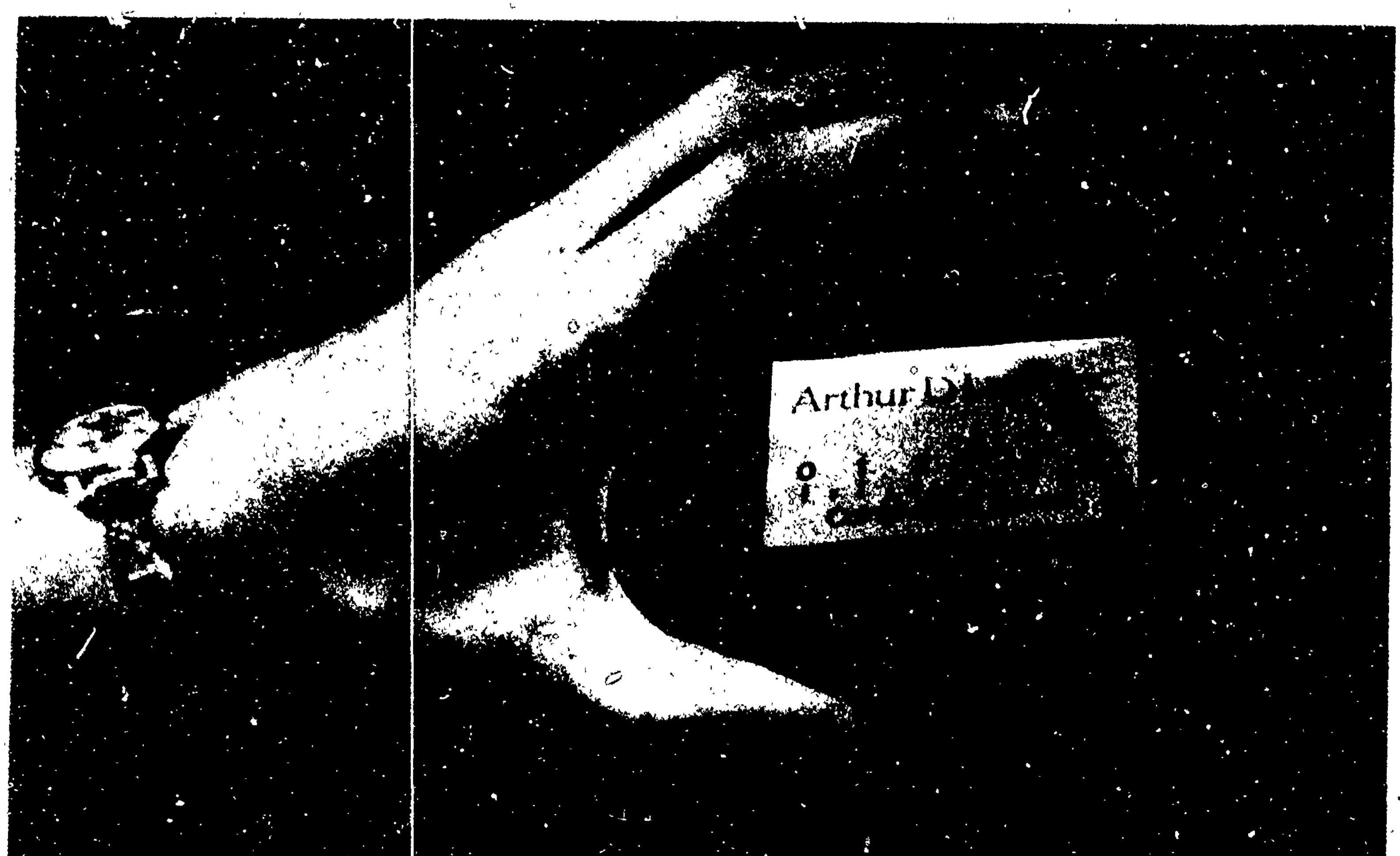


FIGURE 13 Coated and Polished Sapphire Tile

$$\overset{*}{T} = T\frac{(1-R)^2}{(1-R^2T^2)}$$

where T is the bulk transmittance (the reciprocal of the absorptivity times path length) and R is the reflectivity of each surface given by

$$R = \left(\frac{n-1}{n+1}\right)^2$$

For 1/4 inch sapphire with $T \approx .92$ and $n=1.764$, the transmittance should be 0.858. The spectral transmittance of two coated tiles across the visible spectra is given in Figure 14. Within the accuracy of the base line, the observed transmittances agree with calculated value of 85%. Sample 17-1 had a better surface than Sample 13-1 which was polished by a vendor. This may account for the apparent differences between the two samples. The 0.002-inch thick glass coatings do not appreciably affect the intrinsic transmission spectra of sapphire.

In summary, the optical properties of these coated tiles were good enough to demonstrate the feasibility of this concept of surface finishing sapphire tiles. The surfaces did not equal well polished sapphire, but their deficiencies are understood and can be expected to be eliminated with further development.

2. Residual Stress

Residual stresses in the glass coating were evaluated on two of the 1-inch tiles with birefringence techniques. Compressive stresses in the glass ranged from 960 to 1530 psi. Residual stresses were not measured in the coatings on the 4-inch tiles, but they should be nearly the same levels since they experienced essentially the same thermal history.

These results indicate that cracks observed in the 4-inch tiles probably resulted from thermal shock rather than tensile stresses induced by a compressed glass coating. The stress level is too low, and the glass is too thin to break the sapphire by this mechanism.

The residual stresses in the as-received or coated tiles were not evaluated in this program, so it is not possible to comment on the extent to which they may have contributed to tile failures. The thermal cycles to which the tiles were subjected during processing would not be expected to have relieved as-grown stresses to any significant extent. It is evident that as-ground stresses should be minimized, since they would contribute to their susceptibility to thermal shock.

The three parallel cracks in the tile shown in Figure 12 are typical of those observed in several of the 4-inch tiles. These correspond to $(02\bar{2}5)$ planes whose traces intersect the $(1\bar{1}02)$ plane at an angle of 94°. In most cases crystallographic cracks did not propagate through the glass coatings. Randomly propagating cracks, like those shown in Figure 12, generally penetrated the glass coating.

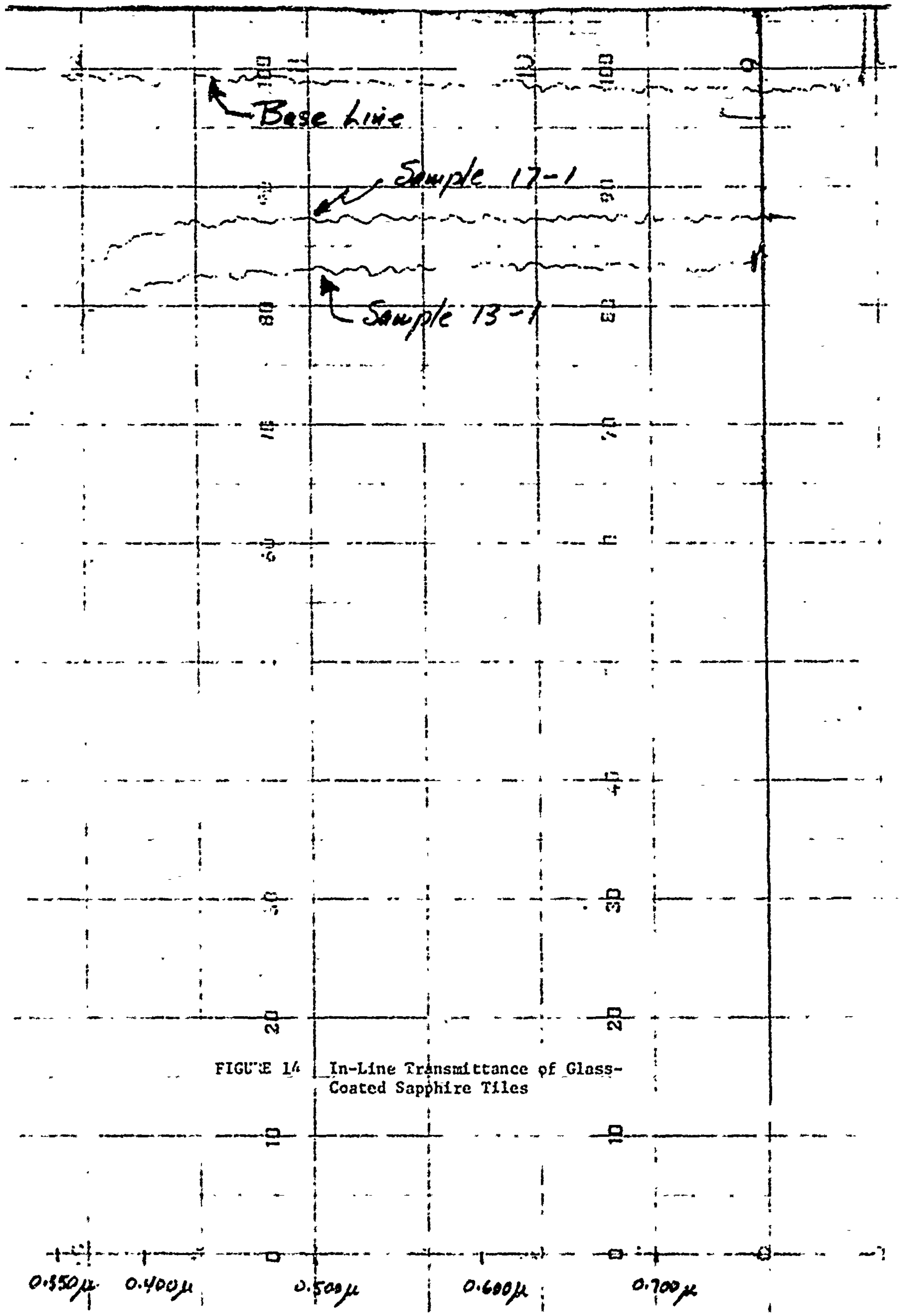


FIGURE 14 In-Line Transmittance of Glass-Coated Sapphire Tiles

It is probably that the glass coatings can be put into substantially higher compression than was achieved in this program. Providing the tiles did not fail due to the induced tensile stresses and the coatings remained adherent to the tiles under the influence of the shear stress induced across the glass-sapphire interface, the strength of the tiles could be improved in a manner analogous to the tempering of glass.

G. Summary and Discussion of Results

The results of this program have shown that glass coating process of sapphire tiles is feasible. Optical quality was not equal to that of well polished sapphire; however, there is no reason to believe that these deficiencies cannot be eliminated with further development of the process.

The processing techniques used in this program revealed several points which must be resolved before the process can in fact be considered a practical alternative to conventional polishing techniques.

Optical quality must be improved. The image distortion in these tiles resulted primarily from localized index variations caused by dissolution of the Al_2O_3 tiles by the glass. It is also possible that vaporization of PbO from the glass may have been a contributing factor, since it has a high vapor pressure and is the constituent which results in the high index of refraction. From the standpoint of optical quality, the glass composition must receive further attention before the optical properties of the finished tiles can be made acceptable.

Loss of tiles by thermal shock was too high to make the process economical. Slower heating and cooling cycles were investigated, but the glass coatings devitrified. Needle-like precipitates formed at the glass sapphire interface and spheriolytes formed on the glass-air surface with slower cooling. The glass composition should be modified to increase its stability and thereby allow slower cooling cycles to be utilized.

It is probable that coating processes based on conventional powder-glazing or vacuum-slumping glass sheets onto the sapphire tiles can be successfully developed. There is no clear advantage either of these processes enjoy relative to the dipping process utilized in this program or more elaborate float glass modifications. They are more familiar to the ceramic industry and thus might be developed more rapidly.

III. COST OF FINISHING SAPPHIRE TILES

We have estimated the cost of finishing sapphire tiles by a glass coating process but have not gone through elaborate cost analyses, since the detailed processing steps we utilized cannot be considered optimum at this time. They are useful for projecting potential finishing costs relative to those used with conventional sapphire polishing and thus determining whether the process is economically viable.

The processing steps utilized in the finishing process developed during this program were: preglazing chemical-polishing, glazing and polishing the glass coatings. All costs are estimated on the basis of unit surface area rather than square inch of finished tile. This distinction has lead to some confusion in the past particularly in projecting sapphire substrate finishing costs to windows since only one surface is finished on a substrate. It also indicates the value of developing individual process steps in which both tile sides are processed simultaneously. For example in our case, both sides of the sapphire tiles were coated in one dipping operation.

For a plant polishing sapphire by the steps utilized in this program, it is estimated that the direct costs for 10^6 square inches per year would be $0.016 per square inch of surface including phosphoric and sulfuric acids, crucibles, furnaces and related controllers and direct labor. The cost of coating the tiles was based on current industry costs for applying a high quality glaze on large quantities of ceramic bodies. Glazing should cost approximately $0.006 per square inch with a well developed process. Polishing costs were derived from the cost of plate glass which is ground and polished as well as vendor estimates for polishing large quantities of glazed tiles by conventional glass polishing techniques. The published selling price of the best quality domestic glass[4] is approximately $0.53 per square foot of glass, ground and polished on both sides, or $0.00185 per square inch of surface area. We made no attempt to isolate the surface finishing costs, but they probably are not greater than $0.001 per square inch of surface are, since both sides are ground and polished simultaneously. A vendor estimated $0.09 per square inch to finish the glazed tiles in large quantities if both sides were finished simultaneously. His costs might be roughly half of this figure or $0.045 per square inch. The differences between these esimated polishing costs (approximately a factor of 50) probably results from the economics of finishing large pieces (typically 48 inches x 72 inches) by highly automated equipment in the case of plate glass and higher quality surfaces from the polishing house, since they normally work to optical tolerances.

Based on these costs, it appears reasonable to expect that the cost of pretreating, coating and polishing sapphire tiles could approach $0.023 to $0.067 per square inch of surface or $0.046 to $0.134 per square inch of tile with adequate process development. Even with admitted uncertainty in these cost estimates, it is clear that the process would result in conventional sapphire finishing. Vendors quote "ballpark" figures of $1.50 per square inch of surface for a high volume, highly efficient sapphire finishing process. Their actual costs might eventually be in the range of $0.70 to $1.00 per square inch. Thus, this surface finishing process could be 5.2 to 22 times less expensive than sapphire finishing processes that are well beyond the current state of the art. The best projected finishing costs for an 8-inch sapphire tile (64 square inches) are $98 to $128 by improved conventional techniques compared to $3.13 to $8.57 for this process. It is obvious that the process can tolerate some tile losses (projected cost of tile blanks excluding finishing is approximately $530) and still remain economically attractive.

IV. CONCLUSIONS

Based on the results achieved in this program, the cost of finishing transparent armor tiles (sapphire) can be significantly reduced by coating rough cut tiles with an index matching glass. It can be reasonably expected that windows can be produced by this finishing process which have optical properties equivalent to those of sapphire. Further process and compositional development will be required to meet the optical requirements, since the glass used in this program attacked to sapphire excessively during the glazing process.

A finishing process based on refiring rough ground, glazed tiles was demonstrated. If surface (coating) flatness can be improved, this process gives smooth surfaces without any of the costly polishing operations. Image distortion, rather than haze was the only deficiency exhibited by tiles finished by this process.

Some chemical polishes used prior to glazing the sapphire tiles produced surfaces which approached the quality needed to meet the optical requirements for windows. These were not pursued during this program, since they involved severe thermal cycles which cracked the 1-inch tiles. Finishing the tiles by chemical polishing warrants further investigation.

From optical property criteria, the feasibility of the process has been demonstrated although further development will be required to equal the quality of well polished sapphire. Loss of tiles during coating by thermal shock must be reduced if the process is to achieve economic viability. It is felt that achieving improved optical properties and less severe thermal cycles are mutually consistent; thus, these goals can probably be reached.

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